

Preliminary investigations on low-pressure laminar plasma spray processing

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Abstract

The usual plasma spraying methods often involve entrainment of the surrounding air into the turbulent plasma core and result in coated materials having relatively high porosity and low adhesive strength. Therefore, exploration of new plasma spraying methods for fabricating high quality coatings to meet the requirement of special applications will be quite important. In this study, an alternative plasma spraying method, i.e. the low-pressure laminar plasma spraying process, is investigated and used in an attempt for spraying thermal barrier coatings (TBCs). Investigations on the characteristics of the laminar plasma jets, feeding methods for the ceramic powder and the formation process of the individual quenched splats have been carried out. The properties of the TBCs sprayed by laminar plasma jet process, such as the adhesive strength at the interface of the ceramic coating/bond coat, the surface roughness and microstructure, are examined by tensile tests and scanning electron microscope (SEM) observations.

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1. Introduction

Ceramic coatings have great potential for many applications due to their good thermal protective feature, high hardness and wear resistance. For example, Al_2O_3 wear-resistant coatings are widely used in high temperature tribological fields [1]. ZrO_2 –8 mol% Y_2O_3 (YSZ) thermal barrier coatings are used as heat-insulating layer on the surface of hot components in gas turbine engines to enhance the thermal efficiency and improve the service life [2–7]. Ceramic-metal composite coatings have been used to reduce friction and wear over a wide temperature range of the hot sections of jet engines and the space shuttle [8,9]. These ceramic-based coatings have been produced by many approaches, such as chemical vapor deposition (CVD), physical vapor deposition (PVD), atmosphere plasma spraying (APS), low-pressure plasma spraying (LPPS), electron-beam physical vapor deposition (EBPVD) and so on. Among these approaches, the plasma spray processing with the turbulent plumes is one of the most effective and widely used [10,11].

Usually the APS processing used in the industry has relative high operating power (30–100 kW) and feed rates (40–100 g/min). Commonly occurring problems with the APS coatings are multi-imperfections and relatively low adhesive strength. The LPPS has the advantage that a controlled atmosphere for deposition of coatings can be created in a limited volume [1,9,12–14]. Due to exclusion of the atmospheric air from the volume, the process greatly reduces oxidation of the powder and substrate materials at elevated temperature and impurity inclusion occurring in the APS processing [15–19]. In low-pressure spraying, the temperature of powder particles can be kept at higher levels than at atmosphere; thus, the injected powder particles can be sufficiently melted. Hence, the LPPS coatings often demonstrate higher adhesive strength and less imperfection.

Sometimes, it is necessary to use special depositing methods for fabricating the high quality coatings [12,20, 21]. For example, in the case of the rapid prototyping of mesoscale mechanical devices using shape-deposition manufacturing methods, the low-power LPPS technology has been used for depositing the ceramic coatings with high adhesive strength and low porosity and the thick metal coatings of relative high bulk mechanical strength at low

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deposition rate (0.1 g/min) and low input powers (2–4 kW). Therefore, developing the potentials of the low-power LPPS processing for depositing refractory materials could be one of the promising plasma spray technologies in the future.

The laminar jet spraying processing may have its place for some special applications. The laminar plasma jets, which can be produced conveniently at relatively low input power conditions (2–15 kW) [22,23], have the features: (i) reducing the entrainment of impurities and eddies of cold ambient gases into the plasma core; (ii) temperature and velocity gradients in the axial direction one or two orders lower than those of turbulence jets; (iii) increasing of the heating time and dwelling distance of the injected particles in the high temperature plasma core. Thus, the laminar plasma jets evidently enhance the heat transfer between the plasma gases and the injected powder particles, and can heat the powder particles more uniformly. Therefore, the laminar jet spraying processing seems promising for spraying high melting point materials in fabrication of the high adhesive strength, low porosity coatings. Furthermore, it demonstrates better operating controllability and much lower working noise compared to the other plasma spray methods.

In this study, a preliminary investigation on the new thermal spraying processing was made and an attempt to produce the TBCs systems was carried out to explore its engineering feasibility and applicational prospects. It includes the production of the laminar plasma jets under atmospheric and low-pressure conditions by using a specially developed plasma generator, the determination of the key factors influencing the characteristics of the plasma jet and the processing conditions influencing the coating structure and properties, and the observations of the molten state of the ceramic powder particles for studying the formation mechanisms of the individual ceramic powder particle splat. It also comprises the tensile tests for measuring the interface adhesive strength and the SEM observations for researching the coating microstructure.

2. Experimental method

2.1. Generation of laminar plasma jets

The stable long laminar plasma jets with high thermal energy density were generated in a vacuum chamber at

pressures lower than 1.3×10^4 Pa. The plasma generator, which consists of a cathode, an inter-electrode insert and an anode, is a home-designed torch. A detailed description of the generation of the laminar plasma jets can be found in Refs. [22,23]. Argon was used as the plasma working gas. The relationships between the plasma jet length and the input conditions were obtained in the low-pressure environment.

2.2. Powder and feeding technology

Three powder-injection sites (Fig. 1) were tested in order to investigate the influence of injection geometry on the laminar plasma jet. The YSZ powders with two different ranges of particle size were tested for depositing the TBCs coatings. One had the small particle sizes up to 25 μm , which showed a bad flow behavior and was difficult to inject into the laminar plasma jet. After some improvements of the feeder, it was possible to inject the fine powder into the laminar plasma cores uniformly and fluently. For comparison, a YSZ commercial powder, which has identical chemical composition and the particle sizes in the range of 25 to 75 μm , was also used for the powder feed testing and the deposition of the ceramic top coatings of TBCs samples. NiCrAlY powder with particle sizes in the range of 5 to 90 μm and concentration of 6% Al and 2% Y_2O_3 was used for deposition of the bond coat. The powder carrier gas was also argon.

2.3. Substrate

The substrate material used was stainless steel 1Cr18Ni9Ti. Before spraying, the depositing surfaces were rubbed with sandpaper of 280 and 600 meshes and blasted by using steel balls of 1-mm diameter. The air pressure for blasting was 6 atm and the time of blasting was 8–10 s. Finally, the substrate samples were cleaned ultrasonically with 5% hydrochloric acid for 10 min and with pure alcohol for 5 min.

2.4. Deposition

Before spraying, the chamber was evacuated to a pressure of 0.2–5 Pa and argon was injected into the chamber until the pressure reached 60–100 Pa. In order to determine

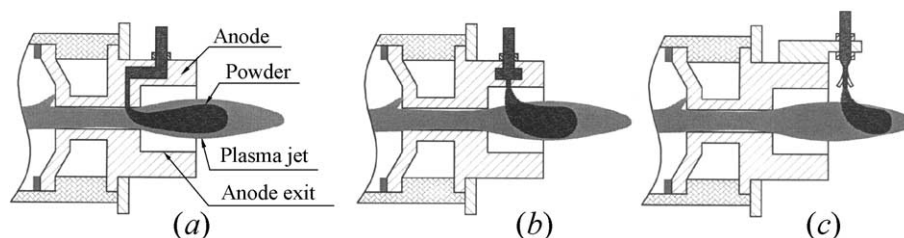


Fig. 1. Three powder injection methods used in this study: (a) inside the anode exit, (b) at the anode exit and (c) outside the anode exit.

the optimum spraying conditions, several operational processing parameters, which included the arc current, gas flow rate, substrate temperature, powder injection site and feeding rate, depositing rate, spraying distance and time, were tested and measured (Table 1). During the spraying, the axis of the laminar plasma jets was normal to the depositing surfaces of substrates and offset 15–20 mm from the center of the sample holder plate (Fig. 2). An infrared pyrometer monitored the substrate surface temperature. The substrate backside was cooled by forced water flow under a pressure of 8 atm.

2.5. Splat formation

Investigations on the morphology of individual splat of two kinds of YSZ powders formed on mirror polished substrates surfaces were conducted in open atmosphere and in low-pressure conditions. To avoid the overlapping of the cooling splats, a low powder-feeding rate was chosen in the both conditions. In the open atmosphere condition, a high moving velocity of the substrate across the cross-section of the laminar plasma jets was adopted, and in the low-pressure condition, a molybdenum plate shutter was used for collecting the individual splats on the smooth substrates. SEM microscopic observations of the states of the collected individual splats were carried out.

2.6. Tensile tests

For measuring the adhesive strength of the TBCs samples, the adhesion tensile tests were conducted on the MTS 810 material test system. The substrate samples were machined into circular plates with dimension of $\phi 18 \times 5$ mm. The spraying materials include the bond coat of NiCrAlY and the YSZ top coating deposited by the laminar jet spraying processing. Before tensile tests, the top surfaces were glued to a steel bar having dimensions of $\phi 18 \times 50$ mm using epoxy at temperature of 100–120 °C for 3 h. Two kinds of samples, of which the ceramic top coats were deposited with the powders of different particle size as mentioned above, were tested, and for each testing condition the average adhesive strength was obtained statistically from five tested samples. Because of limitation in the experimental conditions, such as the small diameter of the laminar plasma jet and the simple design of the depositing equip-

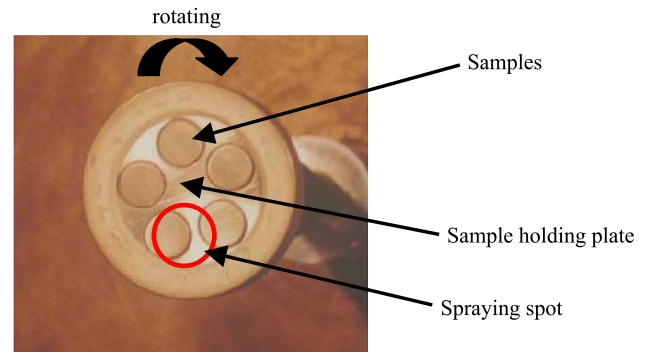


Fig. 2. Sample holder with the TBCs samples and illustration of the spraying spot.

ments, the sample size is limited. The adhesion strength tensile tests were only a preliminary examination on this issue. Further testing in accordance with ASTM C 633 01 standard will be conducted in future studies.

2.7. Microscopic observations

Microscopic characteristics of the coating surfaces, cross section and bond interface were examined by using SEM. The porosity of the ceramic top coating was measured by comparing the difference of contrast of particles on the sample cross section. Surface roughness of the YSZ top coatings was measured with the Taly Surf-5 instrument for the comparison between the laminar jet spraying processing with the fine particle ceramic powder and the APS processing with the common particle ceramic powder.

3. Results and discussion

3.1. Generation of laminar plasma jets

Fig. 3 shows the steady-state appearances of the laminar plasma jets under the low-pressure condition of 60–100 Pa. No apparent surging and whipping of the plasma plume were observed during the process. Effect of input power on the length of the plasma plume is evident. In the wide range of input power from 3.8 to 14 kW, the jet length varied from about 180 mm to much greater than 480 mm, but the diameter of the plasma plumes was maintained at about 25 mm. The profile shown in Fig. 4 indicates the dependence of the length of the laminar plasma plumes on the rate of the axial gas flow with a fixed tangential gas flow rate at input power 12 kW.

As previous researches [10,11] have shown, turbulent plasma jets are a highly heterogeneous system with large temperature and velocity gradients in their short plasma plumes. For example, over a distance of 10 mm, the temperature may drop from 15000 K to almost room temperature and the velocity may drop from 1000 ms⁻¹ to almost zero [10,24,25]. The investigation made by Pan et al.

Table 1

Spraying parameters

Plasma working gas	Ar
Gas flow rate (cm ³ /s)	100 ~ 250
Input power (kW)	5 ~ 12
Powder carrier gas	Ar
Carrier gas flow rate (cm ³ /s)	2.17 ~ 3.33
Chamber pressure (Pa)	60–100
Spray distance (mm)	180 ~ 200
Substrate temperature (K)	500 ~ 700
Sample table rotating speed (r/min)	56

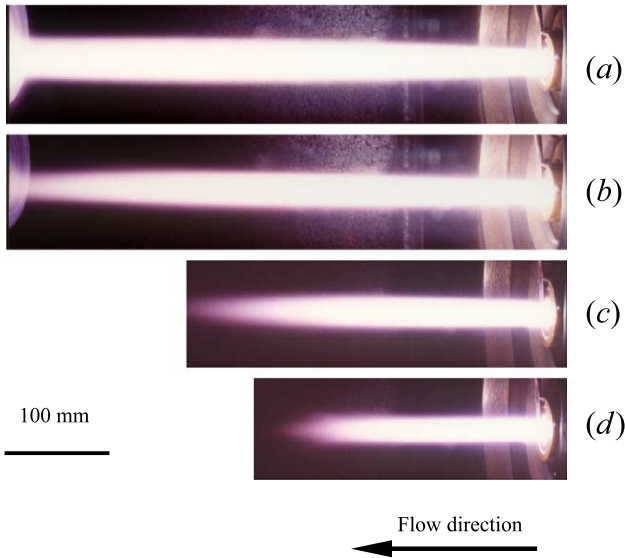


Fig. 3. Appearances of laminar plasma jets at the low-pressure of 1.3×10^{-4} Pa with definite rates of the working gas flow and different plasma power input: (a) 14 kW, (b) 7.8 kW, (c) 5 kW and (d) 3.8 kW.

[22] showed that the temperature gradients of laminar plasma jets with long plasma plumes in the axial direction are about two orders lower than those of turbulent plasma jets, and the temperature distribution in radial direction is more focused, which implies that the laminar plasma jets possess a higher thermal energy density. Otherwise, the ambient gas temperature in the vacuum chamber was quite different with the two plasma states; the experimental measurement shows that it was above 200 °C for the turbulent jets and below 80 °C for the laminar jets. This fact indicates that the thermal energy loss of the laminar plasma plumes to the surrounding atmosphere is much less than that of the turbulent plasma. Hence, it means that the laminar plasma plumes would have higher thermal efficiency in spray process than that of the turbulent ones.

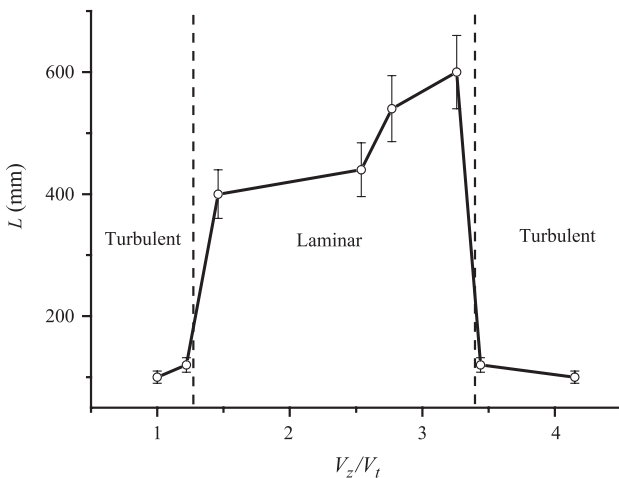


Fig. 4. The laminar plasma jet lengths versus the ratios of the working gas flows. V_z : working gas flow rate in the axial direction and V_t : working gas flow rate in the tangential direction, which is fixed in this tests.

The experimental results indicated that the powder particles injected into the high temperature core of the laminar plasma jets in spraying procedure have lower kinetic energy due to the small rate of the working gas flow. Moreover, the residence time of the particles in the laminar plasma plumes was much longer than that in the turbulent plume. Hence, more efficient heat transfer between the powder particles and plasma gas can take place, which is favorable for melting of the refractory materials. Furthermore, the lower kinetic energy of powder particles would limit the splashing phenomenon of the splats when the powder particle droplets impacted the substrate. All these would be helpful for high quality coating deposition by the laminar jet spraying processing.

3.2. Powder feeding

Fig. 5 shows the laminar plasma jet appearance at several powder feeding rates with different injection sites in the optimizing range of the carrier gas flow rate, 130–200 cm^3/min . In this figure, pictures (a)–(c) correspond to feeding rates with the common size YSZ powder of 7, 14 and 23 g/min in the low-pressure conditions with the injection site outside the anode exit (Fig. 1c). Picture (d) shows the atmospheric-pressure feeding condition of the small particle size YSZ powder at feeding rate of 7 g/min outside the anode exit (Fig. 1c). Picture (e) shows the low-pressure spraying condition with the small particle powder at feeding rate of 14 g/min inside the anode exit (Fig. 1a). In all

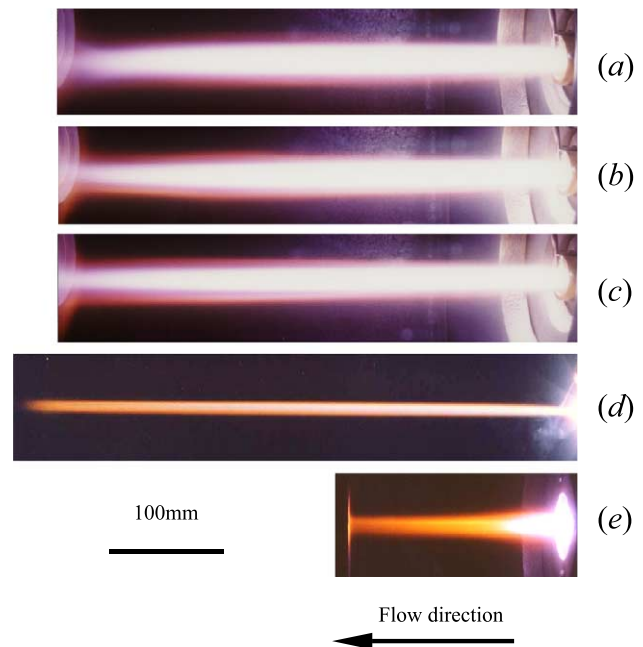


Fig. 5. Appearances of the laminar plasma jets at the low-pressure of 1.3×10^{-4} Pa with different plasma power input and powder-feeding rates; (a) 10 kW, 6–8 g/min; (b) 10 kW, 13–15 g/min; (c) 12 kW, 22–24 g/min; (d) 9 kW, 6–8 g/min (at atmosphere conditions) and (e) 8 kW, 6–8 g/min (depositing the YSZ powder at low pressure).

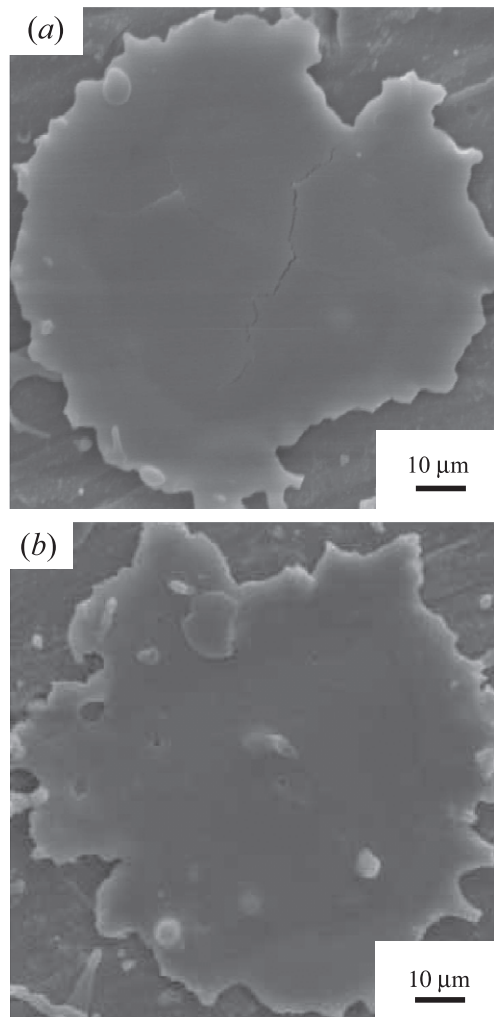


Fig. 6. Shape and morphology of the individual ceramic splats sprayed on the stainless steel substrate kept at temperature 400–500 °C at atmospheric pressure.

situations, the deposited materials reached about 28–30% of the injected amount. It has been observed that for the powder injection sites a and c, the plasma jets can be kept laminar and the powder can be injected into the high temperature core of the plasma jets conveniently. Hence, the two injection sites are suitable for the laminar jet spraying processing. However, for the injection site b, the stable status of laminar plasma jet could only be maintained for about 30 s at the powder feed rate 14 g/min, that is, at that time a change of the plasma plume from laminar to turbulent occurs, which indicates that the powder injection position has an important influence on the state of plasma jets.

The experimental results indicate that the powder-feeding rate also influenced the state of plasma jets. When the feeding rates were lower than a critical value about 18 g/min, the steady-flow of the laminar plasma jets was observed for two injection sites, inside the anode exit (Fig. 1a) and outside the anode exit (Fig. 1c). Above the critical value, the steady state

of the laminar plasma jet could not be kept for the powder injection site inside the anode exit. At this time, the surging and whipping of the plasma plums were observed frequently. However, the phenomena were not observed for the injection site outside the anode exit even when the feeding rates reached 23 g/min and the feeding time reached 20 min (Fig. 5c). In these tests, the injection of powders into the high temperature plasma core was easily accomplished under the low-pressure conditions because the laminar plasma jet has a larger diameter about 25 mm, whereas it was difficult at atmospheric conditions because its diameter was only about 5 mm (Fig. 5d). No evident effect of the powder particle size on the state of the plasma flow was observed.

3.3. Splat formation

Preliminary study on the molten conditions of individual powder particle was carried out. The morphologies of the individual splats deposited on the smooth substrate at temperatures 400–500 °C in both the atmospheric pressure and the low-pressure are shown in Figs. 6 and 8, respectively. The depositing distances are 120 mm (Fig. 6a) and 150 mm (Fig. 6b) at atmospheric pressure, and 170 mm (Fig. 7a) and 210 mm (Fig. 7b) in low-pressure, respectively. These values represent the optimum ranges of depositing distances in both conditions.

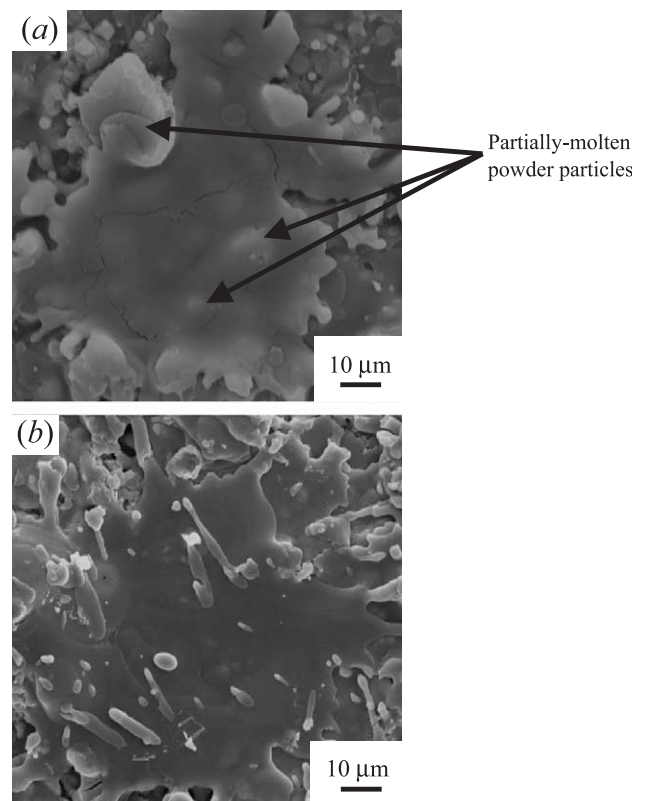


Fig. 7. Shape and morphology of the individual ceramic splats deposited on the stainless steel substrate maintained at temperature 400–500 °C at low pressure.

It is well known that the coating property is closely related to the molten state of the ceramic powder particles and the impact velocity of the droplets on the substrate [11]. The splats morphology (Fig. 6) indicates that the common ceramic particles have been completely melted in the spraying process. An evident feature of the splats is that they have compact pancake shapes without splashing structure, which is clearly related to the properties of the laminar plasma jets as mentioned above. During the depositing process, the lower velocity attained by powder particles corresponds to lower kinetic energy, which makes the impacting force of the droplets on the substrate evidently reduced to avoid shattering of the droplets. Moreover, the longer heating time and moving distance of the powder particles in the laminar plasma jets provided better heating and reduced material viscosity. Therefore, the droplets could spread out on the substrate steadily and uniformly, resulting in the reduced splat thickness. Another feature of the splats is that less cracks formed in solidification, which is beneficial to the fabrication of high-density coatings and higher interlamellar strength.

From the micrographs of individual splats of the small particles size powder in low-pressure conditions (Fig. 7), it can be seen that some partially molten powder particles (as indicated by the arrows in the figure) exist in these isolated splats, but such particles were not observed in atmospheric deposition (Fig. 6). The observation indicated that the laminar plasma jets in atmospheric pressure are more suitable for the melting of the ceramic materials like the YSZ powder than in the low-pressure condition.

The investigation on flow behavior of powder materials [3,11,26] showed that the typical distributions of the powder particle size for the plasma spraying are in the range 10–45 μm for ceramics. In the spraying process using the ceramic powder of particle size below 20 μm , the collisions among the powder particles and with the pipe wall of the powder feeder are probably responsible for the particle trace divergence when they were injected into the plasma core. At present, perhaps the dispersion of particle trace results in the partially molten particles deposited in the individual splats. The existence of the partially molten droplets may be one of the reasons of reduced adhesive strength of the ceramic/bond coat interface as seen in the following section.

3.4. Adhesive strength

The full view of the separated surfaces at the interface of the ceramic coatings/bond coat of the tensile testing samples is shown in Fig. 8. Figure (a) is the exposed surface of bond coat and figure (b) is the matching ceramic surface of the ceramic coat. The macroscopically homogeneous fracture surfaces suggest that the samples were subjected principally to simple tensile loading in the testing process and the stress states at the cracking interface approximately complied with one-dimensional loading conditions.

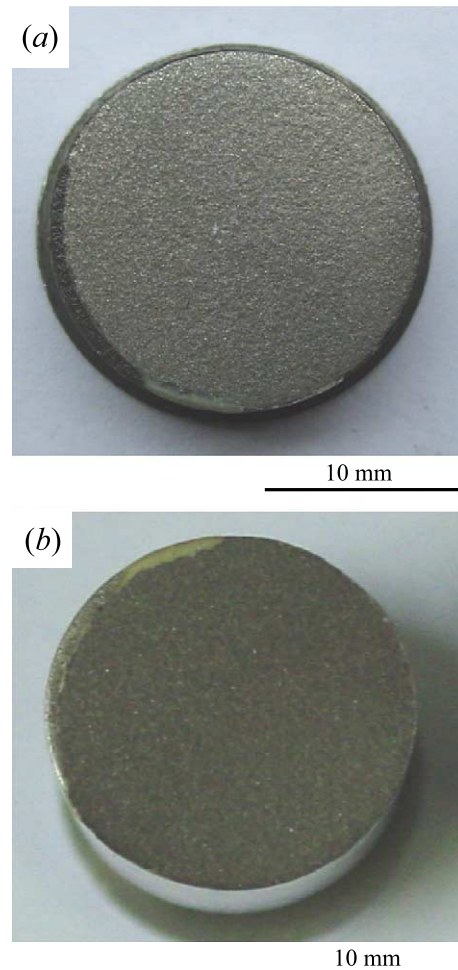


Fig. 8. The fracture surfaces of the TBCs samples after tensile testing: (a) morphology of the BC surface and (b) matching ceramic surface of TBCs.

As shown in Fig. 9a, the adhesive strengths at the interface of the ceramic/metal are above 40 MPa for all testing samples sprayed with the common particle size powder. The maximum adhesive strength reaches 64.23 MPa and the average value is 51.12 MPa approximately, which illustrates that the laminar jet spraying processing can deposit the coatings with high adhesive strength. For the testing samples deposited with small particle size powder, two of the measuring results of the adhesive strengths are below 40 MPa. The maximum and average values of the adhesive strength are 56.14 and 44.64 MPa, respectively (Fig. 9b). The low interface toughness indicates that the flowing behavior and the particle size of the ceramic powder influence considerably the attaching strength. The result is an agreement with the situations of the fine powder feeding in depositing process and the microscope observations of the individual splats as mentioned above.

3.5. Microscopic observations

Fig. 10 is an SEM view of the polished cross section of the TBC system samples deposited with the small particle

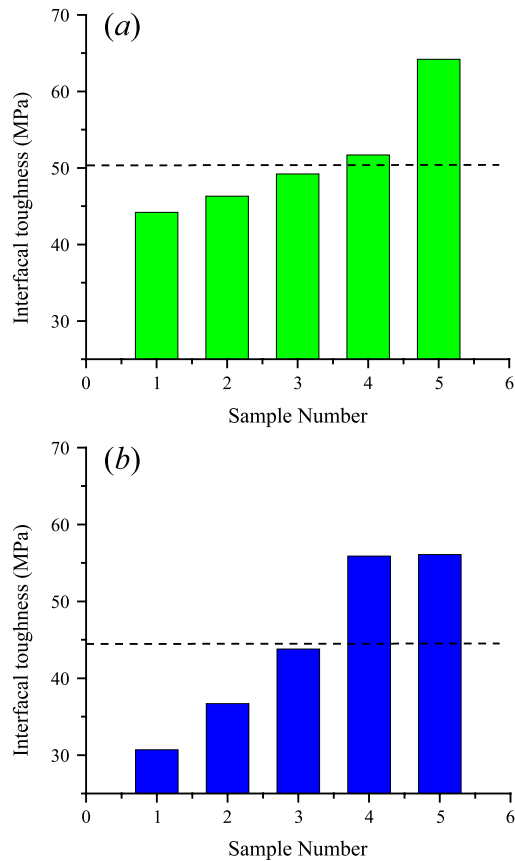


Fig. 9. (a) Adhesive strength of the samples deposited with the common particle size powder and (b) samples deposited with the small particle size powder.

size powder. Note that no highly undulations at the interface are observed. These undulations, often appearing in plasma spray coatings, are favorable for adhesion of the interface by increasing the mechanical anchoring. However, they often induce imperfection and micro-cracks and bring about tensile stresses normal to the interface, which cause the eventual failure of TBCs [27]. The smooth interface greatly diminishes the probability of imperfection growing and magnitude of the tensile stresses and improves the adhesive mechanism of chemical bonding and the ability against thermally grown oxide. All of these are beneficial to improving the TBCs properties and extending their service life.

Fig. 11 shows an enlarged view of the fracture surface of TBC ceramic layer. Four features can be seen: (i) the ceramic top coatings have the typical lamellae microstructure of plasma spraying; (ii) the thickness of each progressively deposited layer is only about 3 μm ; (iii) fewer non-molten and partially molten powder particles are noticed (as shown by the arrows in the figure); (iv) the average porosity measuring in the ceramic top coating is only 1.37%. These features mean that the laminar jet spraying processing can be utilized to deposit ceramic coatings with high density.

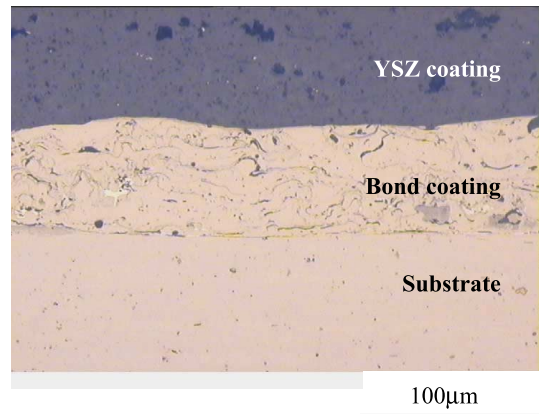


Fig. 10. Polished sectional micrograph of the TBCs system fabricated by the laminar jet spraying processing.

Fig. 12a and b shows the SEM surface views of the ceramic top coatings deposited with small/common particle size powder, respectively. It can be seen that the overall uniformity of the coating sprayed with the common particle powder is better than that with the small particle powder, in which more area consisted of the non-molten and partially molten powder particles. In Fig. 12c and d, the corresponding enlarged SEM views of the surfaces are given for the top coatings. The similar phenomena, such as, no apparent splashing of the quenching splats and existence of the partially molten ceramic particles in the splats of small particle powder were also noticed. These figures also show the lamellae microstructure of the typical plasma spraying coatings and the pores (indicated by the arrows) in the ceramic coatings, which are favorable to the increasing of strain tolerance of the coatings.

When the powder-feeding rate is below 6 g/min, the depositing efficiency can be above 35%, and the coatings microstructure shows evidence of full melting of the ceramic powder. Fig. 13 shows the SEM view of the exposed surface at the separated interface of the ceramic coatings in contact

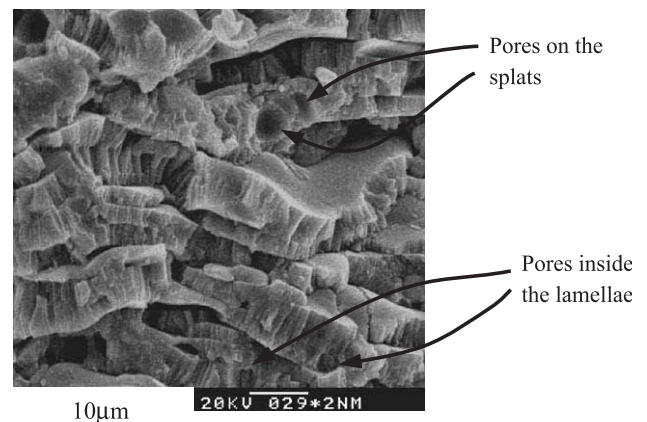


Fig. 11. SEM view of the fracture cross section of the ceramic top coatings in the TBCs system. A few pores denoted by arrows can be found.

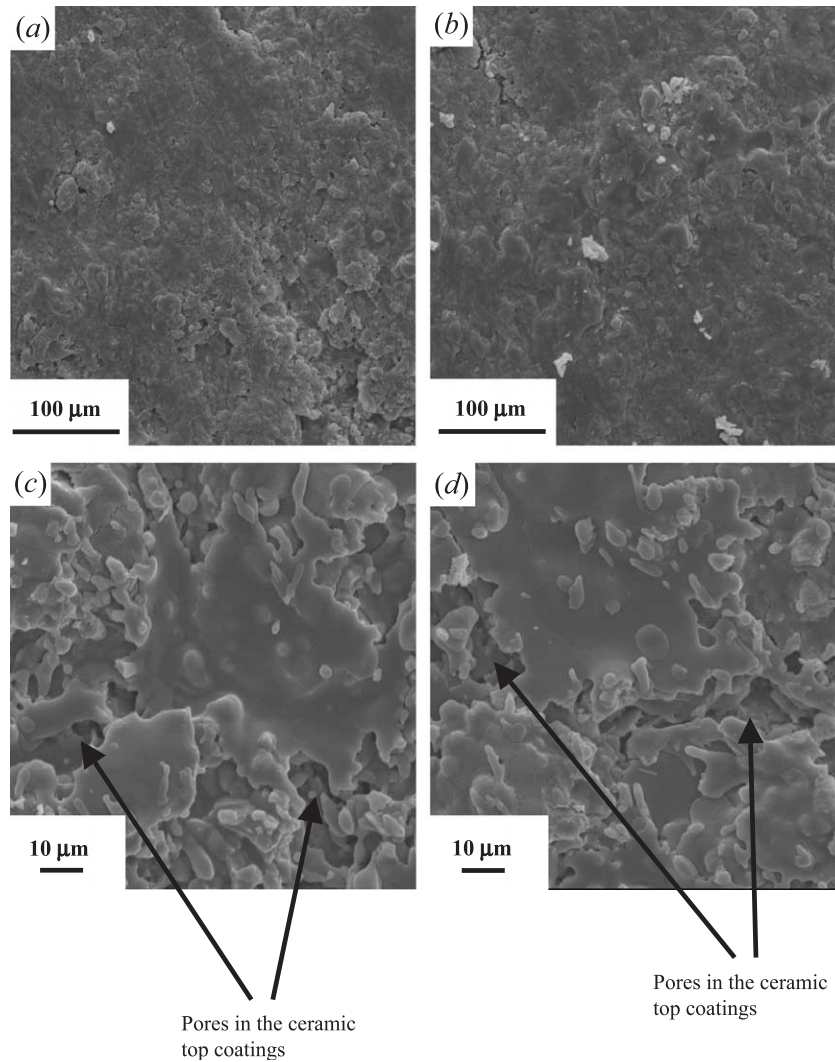


Fig. 12. (a) SEM views of the ceramic top coating surface of the samples deposited with small particle size powder; (b) that of the common particle size powder; (c) and (d) enlarged SEM views of the ceramic top coatings surfaces deposited with the small and common particle powders, respectively. The arrows denote the pores appearing at the joints of the overlapping ceramic splats.

directly with the steel substrate. The surface marks match the microscopic morphology of the substrate surface very well, which shows that the powder particles could attain sufficient heating to form the fully molten droplets before depositing on the surface. Besides, the molten droplets in flight could gain moderate velocities corresponding to impacting forces, enabling themselves to form mechanical anchoring with the substrate surface in the solidifying interval. This indicates that close joining between the ceramic splats and the substrate surface had taken place.

Fig. 14 shows the measured surface roughness of two kinds of samples. One is coated with the small particle powder by laminar jet spraying in this work, and another is sprayed with the commercial YSZ powder by APS. The coating surface from the small particle powder shows lower roughness, and its relative value, R_a , is about 2.7 μm. In contrast, the APS sample surface possesses higher roughness

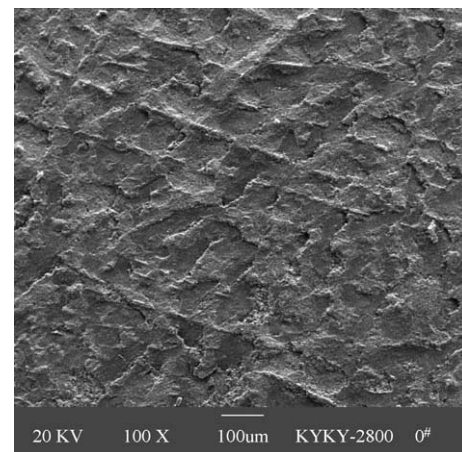


Fig. 13. SEM view of the exposed surface at the separated interface of the ceramic top coating in contact directly with the steel substrate.

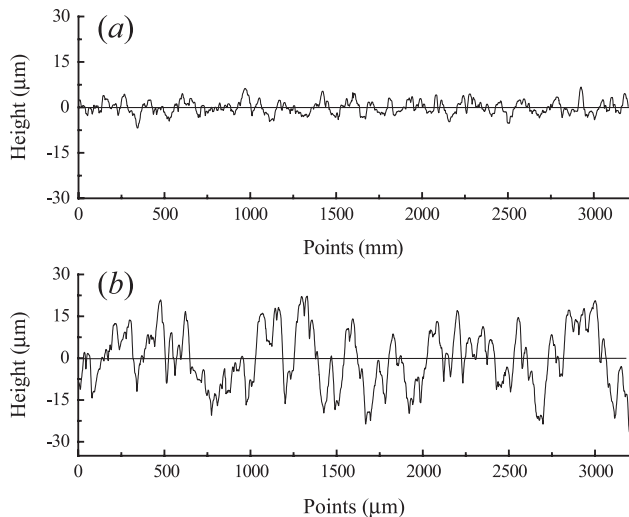


Fig. 14. Measured specimen surface roughness: (a) with the small particle powder by low-pressure laminar plasma spraying in this work and (b) from traditional atmospheric plasma spraying.

ness, and its R_a is $7.3 \mu\text{m}$. The results on the surface roughness are consistent with the TBC microstructure of the small thickness and undulation of the splat layer.

4. Conclusions

Steady laminar plasma jets can be produced in a controlled atmosphere. The lengths of the plasma plumes are related both to the injection pattern of the working gas and the input power. Laminar jet spraying processing has good utilization of the thermal energy of plasma gas while maintaining a moderate kinetic energy of the powder particles, which is favorable for producing coherent quenching splats without splashing and micro-cracks, yet with sufficient adhesive strength at interface by mechanical anchoring. The YSZ powders with different particle size were sprayed to fabricate ceramic coatings in the input power of 8–12 kW. The coatings possess not only the typical lamellae structure, but also thin deposited layer and dense microstructures. The powder feeding technologies were compared in low/atmospheric pressures. Powder feeding inside and outside the anode exit at the feeding rates of 14 and 23 g/min, respectively, are optimum conditions for effective heating and melting of powder particles without affecting the laminar plasma plume states. The adhesive strengths at ceramic/metal interface have reached the common levels of the APS coatings and the surface roughness of ceramic coatings is only a third of them. In addition, the argon laminar jet spraying processing does not induce the phase structure change of the raw YSZ powder materials. These studying results show the technical feasibility of the laminar jet spraying processing in the thermal spraying fields, especially in situations requiring low input power and low deposition rate.

More complete investigations are needed on the deposition process of the ceramic coatings at atmospheric and low-pressure conditions. One important aspect deals with the powder-feeding problem. Successful deposition of the ceramic powders requires their feeding into the high temperature cores of the small-diameter laminar plasma jets and reduces the partial- or non-molten particles in the coatings. Studies on the life and failure mechanisms of the TBC samples upon thermal cycling are also needed to examine the resistance to the thermally grown oxide of the TBCs materials and to prove the advantages of laminar jet spraying in the thermal spray technology.

Acknowledgements

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